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Structure and the Physico-Mechanical Properties of the Ceramic Coatings Obtained by the Cumulative -Detonation Device

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Dense, with good adhesion to the substrate, hard, wear-resistant coatings from the powder of Al_2O_3 were obtained on the surface of the steel (STE255) by using the cumulative-detonation device. The results of investigations of the structure and physico-mechanical properties of the coatings by using scanning, optical microscopy, X-ray phase analysis, microhardness and tribological tests are presented. It was found that optimization of plasma spraying to helps reduce the porosity of coatings of Al_2O_3 less than 1 % and to increase the hardness of them to 1250 $HV_{0.3}$. The tribological investigations have shown that the coatings of Al_2O_3 significantly increase the wear resistance of the sample STE255 and provide a low ability to wear out the coating.

Keywords: Aluminum oxide, Cumulative-detonation technology, Microhardness, Wear resistance.

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1. INTRODUCTION

The ceramic coatings from the powder of Al_2O_3 are usually used to protect the details of which work in the hard conditions. They show a high resistance to corrosive and oxidizing environments and provide protection for details of which work at high temperatures. In addition, powders based on aluminum oxide have a low value, which determines the cost-effectiveness of their application. A new direction in this area is modification of surface by plasma-detonation technology that allows obtaining the ceramic coatings with a definite composition and thickness of reduction of the cost, which determines the cost-effectiveness of their application. Plasma-detonation technology is based on the use of pulsed plasma jets, accelerated by the electromagnetic field up to speed 5000...8000 m/s [1]. All this makes it possible to obtain high quality coatings of ceramic and metal on metal substrates [2]. The aim of this work was to study the structure and the physical and mechanical properties of high-quality coatings of Al_2O_3 obtained by detonation-cumulative device.

2. METHODS OF SAMPLE MANUFACTURING AND ANALYSIS

The cumulative-detonation technology and equipment is created for deposition of nanocomposite powder coatings on samples. The device provides a high velocity of the powder materials (>2000 mm/min) without its overheating. Productivity was 0.72÷1 kg/hour, gas mixture consumption (C_3H_8+O_2) – 5.08 m^3/hour, frequency – 20 Hz, the nozzle section to a specimen - 60 μm. Powder of Al_2O_3 (99.5%) with fraction 5.50 -62 μm was used (see. Fig. 1). Specimens of steel STE255 were used as a substrates. A uniform coating ~ 200 μm thick was deposited on the specimen's surface.

Morphological and compositional characterization of powder of Al_2O_3 and surface of ceramic coatings were performed by electron ion microscope Quanta 600 FEG equipped with integrated microanalysis system Pegasus 2000 (SEM-EDS). The microhardness of coatings was determined on transverse sections of system "coating/substrate" by an automatic analysis system microhardness DM-8 according to the method of micro-Vickers. X-ray diffraction studies of coatings were performed on a diffractometer ARL X'TRA company Thermo Scientific.

The evaluation of wear resistance of coatings was carried out by the methods of tribometer using the an automated machine friction (Tribometer, CSM Instruments, Switzerland) under the standard scheme of test "disc/ball" on the air under a load of 6 N. The porosity of the coatings from the powder of Al_2O_3 was determined by metallographic method using an optical inverted microscope Olympus GX51.

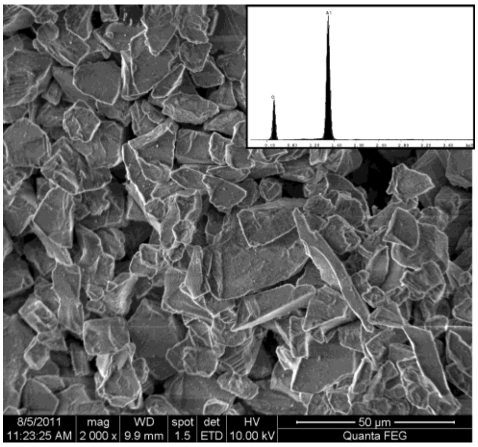

Fig. 1 – Morphology and elemental composition (energy dispersive spectrum) of Al_2O_3 powder (SEM)

3. RESULTS AND DISCUSSION

Electron microscopic investigation of transverse sections of the «coating/substrate» has shown that the coating of powder of Al_2O_3 characterized by the alternation

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of compressed flakes and deformed discrete particles of the oxide. Coating formed by stacking a set of deformed coherent particles with different temperature, velocity and mass (see. Fig. 2).

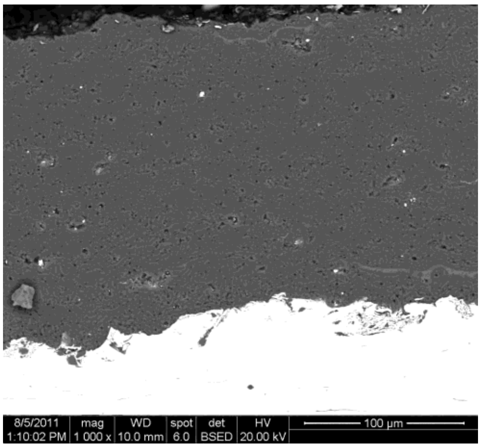
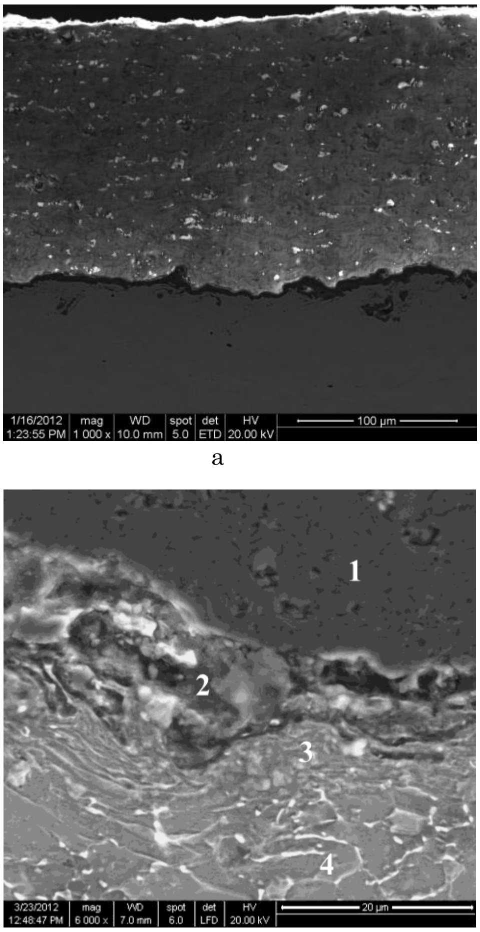


Fig. 2 – Microstructure of transverse sections of the sample with coating of Al_2O_3

Thick coatings with good adhesion to the substrate and the low porosity less than 1% obtained by plasma-detonation device from the alumina powder (see. Fig. 2). The quality of coatings was achieved by the presence of α-Al_2O_3, its share to 67% (see. Fig. 3). Analysis of the structural features of the interface "coating/substrate" has shown that the plasma-detonation powder coatings have three visually distinguishable zones (substrate, transition layer and main coating) (see. Fig. 4a). It should be noted that the elemental composition of the transition region from point to point remains almost unchanged. The main components of the matrix are the Al and O, and their concentration at different points changes insignificantly. Impurity elements in the transition region of the substrate are the elements of the substrate Fe, Cr, C, Na and Si. The presence of impurity elements in the coatings in the transition region has been caused by mixing of detonation gases, air and sputtered particles of the substrate.

Analysis of the structure of the interface "coating/substrate" after etching has shown that the coating has four visually distinct zones (see. Fig. 4a). Content of aluminum in the transition region in the point 2 was 76%, in the point 3 - 49% (see. Table 1). Therefore, the material of boundary was a mix of elements of the substrate and coating.



a

b

Fig. 4 – Structure of Al_2O_3 coating (SEM)

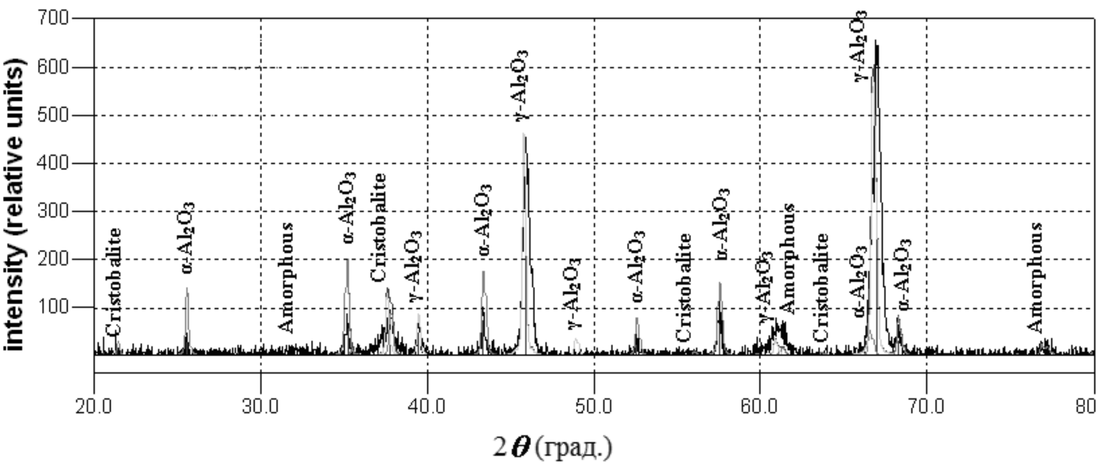


Fig. 3 – Results of X-ray analysis of Al_2O_3 coating

Table 1 – The elemental composition of the material of the boundary and sample of the surface (Fig. 4a)

Point	Content of the elements, at.%				
	Al	O	Fe	C	Si
1	54.02	45.98	-	-	-
2	76.13	22.48	-	0.96	0.43
3	49.99	24.21	20.33	3.98	1.16
4	0.20	4.37	87.52	5.65	0.62

At the boundary between the transition zone and the main coating (point 3) the concentration of atoms of aluminum was about 50 wt.%, and oxygen ~24 wt.%. It was established that the content of iron in the transient region is 20 wt.% what can be attributed to its mass transfer from the substrate.

It was found the presence of iron, metallic aluminum and intermetallic AlFe on the border between the coating and substrate. This favors the relaxation of stresses arise during the deposition of coatings [3]. Therefore, the iron should be considered as an impurity, which is a favorable effect on the mechanical properties of coatings.

The content of the carbon in the ceramic coating of the Al_2O_3 is principled. The presence of significant amounts of carbon in the coating leads to a deterioration of service characteristics. For example, the presence of carbon reduces the density of the coating and the formation of pores, which significantly decreases the corrosion resistance. Also, the presence of carbon in the coatings affects the cohesion between layers of sprayed coatings, as well as the adhesion between the coating and steel substrate. The interaction of carbon with iron formed brittle phase, which leads to the appearance of cracks on the boundary.

Thus, to improve the performance properties was selected regime of the deposition of the coating in an oxidizing atmosphere (with excess oxygen). Oxidizing atmosphere also prevented the hydrogenation coatings, as it is known that alumina adsorbs hydrogen well in a heated state. Thus, the results of microanalysis of the transition layer and separate sections covering confirm the mixing of the coating material - Al_2O_3 with the substrate material, indicating that a strong connection of the resulting system "coating/substrate" interface.

The microhardness of coatings was determined from transverse sections. It was found that the hardness of coatings Al_2O_3 was 1500 ± 25 $HV_{0.025}$, which indicates that the homogeneity of sufficient density but adjacent layers of the coating. It should be noted that the hardness of the coating of 4.3 higher than the hardness of the substrate material STE255 – 346 ± 5 $HV_{0.025}$. It should be noted no significant spread of values of the microhardness of coatings from point to point (see. Fig. 5). This could mean that coatings have the phase and structural homogeneity. The microhardness of coating layer is stable and its fluctuations did not exceed 5%. The coatings were sufficient plasticity, as evidenced by the absence of cracks in the corners of the prints at a load of 25 g. Cracks were appeared only at a load of 300 g (see. Fig. 6). At a load of 300 g of the microhardness values are somewhat lower. This phenomenon is explained by the elastic reduction [4].

The microhardness of the substrate of steel STE255 under the coating was changes to a depth of 200 μm from 550 $HV_{0.025}$ to medium hardness of the substrate material -346 $HV_{0.025}$. The figure 7 demonstrates that the microhardness of the substrate decreases by 1.5 times at a depth of 15 microns from the boundary "coating/substrate" and reached the minimal value at a depth of 180 microns. It means explains the presence of work hardening near the boundary with the surface, was formed as a result of impact in the process of abrasive blasting and coating.

The microhardness of coating was measured from the surface to the substrate gradually decreases from the surface of the substrate from 1250 to 900 $HV_{0.3}$. This decrease is due to the mechanism of formation of layered coating and heating below the underlying layers. These conditions are the result of the formation of a soft plastic and aluminum oxide phases.

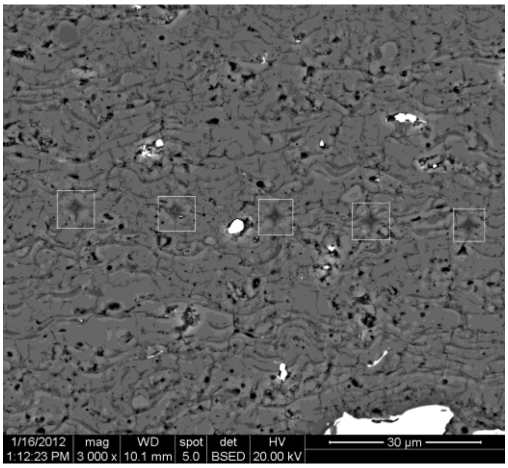


Fig. 5 – SEM image of microstructure of Al_2O_3 coating with measuring microhardness ($HV_{0.025}$)

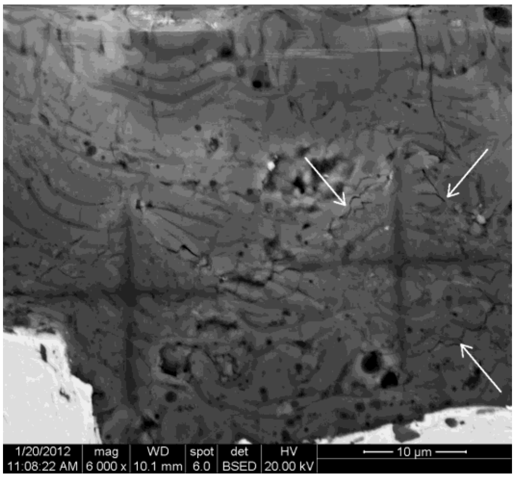


Fig. 6 – SEM image of microstructure of Al_2O_3 coating with measuring microhardness ($HV_{0.3}$)

Wear resistance tests performed on air at load of 6 N, linear speed is 15 cm / sec, the radius of curvature of 5 mm of wear, sliding distance of 1200 meters. From the data of table 2 shows that the investigated coating significantly increases the wear resistance of the sample STE255, also provides a low ability to wear out the coating.

Investigation of the topology of friction surface coating shows that the initial friction coefficient of coated steel was 0.038, which may be due to the high roughness of the sample. Found that despite the increase in counterface of wear, track wear and tear, however, without destroying the coating, which is associated with good wear resistance of the coating.

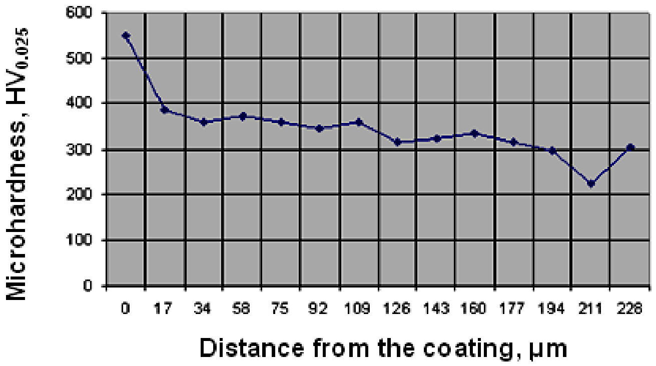


Fig. 7 – The microhardness of the substrate

Table 2 – Tribological characteristics

Sample	The coefficient of friction (μ)		The wear factor, $\mu m^3 \cdot N^{-1} \cdot m^{-1}$	
	initial	test	counter body (x10^{-5})	sample (x10^{-5})
Steel (STE255)	0.204	0.674	0.269	35.36
Steel/Al_2O_3	0.038	0.959	1.61	19.39

4. CONCLUSION

Ceramic multifunction dense coatings from the powder of Al_2O_3 were obtained by the cumulative detonation technology. The microhardness of coatings decreases smoothly from the surface of the substrate from 1250 to 900 $HV_{0.3}$. In spite of the high hardness of the coatings, they are sufficient plasticity, which confirms the absence of cracks in the corners of indents at a load of 25 g.

Analysis of the transition layer "coating/substrate" interface was showed that the layer is well adjacent to the substrate. Is formed transition layer, consisting of the dissociation products of elements of the substrate and the oxide. The hardness of the substrate layer is adjacent to the coating, increasing to 550 $HV_{0.025}$ at a hardness of the basis 340 $HV_{0.025}$.

Optimization of plasma spraying to helps reduce the porosity less than 1 % and to increase the hardness of the coating to 1250 $HV_{0.3}$. When spraying agglomerated powders, micron particles collected from the ceramic coating hardness reaches 1350 $HV_{0.3}$ using as a fuel gas of hydrogen and 1000 $HV_{0.3}$ using propane.

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